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- Bleach activator compositions manufacture and use thereof in laundry compositions.
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Description

The present invention relates to bleach activator compositions. More particularly, it relates to bleach activator compositions prepared by spray drying for use in or with a detergent and/or bleach composition. The bleach activator compositions have excellent storage stability, physical strength and water-dispersibility, as well as simplified processing and reduced segregation in a full detergent or bleach product. The invention also relates to a process of making the compositions and also to laundry compositions containing water-soluble bleach activators.

It is well known that peroxygen bleaching agents, e.g., perborates, percarbonates, perphosphates and persilicates, are highly useful for chemical bleaching of stains found on both colored and white fabrics. Such bleaching agents are most effective at high wash solution temperatures, i.e., about 60°C to 70°C. In recent years, attempts have been made to provide bleaching compositions that are effective at lower wash solution temperatures. In consequence, there has been much industrial research to develop substances generally referred to as bleach activators, that render peroxygen bleaches effective at bleach solution temperatures below 60°C. These low temperature bleaches are useful in a variety of products intended for use under machine or hand-wash conditions, e.g., additive, pre-additive or soak-type laundry compositions as well as all-purpose detergent compositions.

A bleach activator can generally be described as an organic peroxy acid bleach precursor which in the bleaching solution reacts with an inorganic peroxygen bleaching agent with release of the organic peroxy acid. Many such bleach activators are known in the art, most of which contain perhydrolysable N-acyl or O-aryl residues. Examples of these include succinic, benzoic and phthalic anhydrides, tetracetylethylene diamine and tetracetylglycoluril, all of which are water-insoluble materials to varying degrees. See FR—A—2,148,203 for a description of such materials.

Water-soluble bleach activators are also known in the art and are described in detail in, for example, US—A—4,412,934 and EP—A—0120591. These activators have a number of advantages over water-insoluble materials including rapid dispersibility in wash or bleach solution and also the ability to exhibit surface bleaching. On the downside, however, water-solubility greatly exacerbates activator stability problems resulting from interaction with moisture and peroxygen bleaching agent during prolonged storage in the bleach or detergent composition.

It is also known in the art to incorporate bleach activators in a detergent slurry for spray-drying (see GB—A—1540832 and FR—A—2307869) whereby the resulting detergent composition comprises the bleach activator homogeneously distributed therein. EP—A—0070474 also relates to the spray drying of bleach activators. In the case of water-soluble bleach activators, however, it is found that the conventional crutching and spray-drying process results in essentially complete degradation of the activator.

It has now been found, however, that water-soluble bleach activators can be successfully crutched and spray-dried by incorporating an inorganic hydratable material in the crutcher slurry mix and adjusting the pH of the slurry to within the range from 3 to 8 prior to spray-drying. Moreover, the subsequent granule is found to combine excellent stability characteristics when stored in a detergent or bleach composition, as well as improved dispersibility on addition to the wash or bleach solution.

According to the present invention, therefore, there is provided a bleach activator composition in the form of spray-dried granules having an inner core comprising organic peroxyacid bleach precursor and a surface coating comprising a water-soluble or water-dispersible, inorganic hydratable material, characterised in that the organic peroxy acid bleach precursor is water-soluble and wherein the composition has a pH (1% aqueous solution) in the range from 3 to 8.

The invention also relates to a process of making a granular bleach activator composition comprising forming an aqueous slurry of a water-soluble organic peroxyacid bleach precursor and a water-soluble or water-dispersable inorganic hydratable material, adjusting the pH of the slurry (1% solids) to within the range from 3 to 8, preferably from 3.5 to 7 and subjecting the slurry to spray-drying.

In its composition aspect, the compositions of the invention take the form of granules having an inner core comprising water-soluble bleach precursor encapsulated or substantially enclosed within a coating of water-soluble or dispersible inorganic hydratable material. In preferred embodiments, the inner core exists as an essentially spherical droplet or aggregation of spherical droplets of the bleach precursor.

The outer coating on the other hand comprises a water-soluble or water-dispersible inorganic hydratable material which in preferred embodiments, consists majorly (at least 50%, preferably at least 75%) of one or more acid salts having a pK_a in the range from 1 to 9, preferably from 2 to 7, more preferably from 3 to 6.5. Suitable inorganic hydratable materials include disodium dihydrogen pyrophosphate, tetrasodium monohydrogen tripolyphosphate, trisodium dihydrogen tripolyphosphate, sodium bicarbonate, acidified pentasodium tripolyphosphate, acidified borax and acidified aluminosilicates and mixtures thereof. Strong acid hydratable salts such as sodium sulphate and magnesium sulphate can also be incorporated in minor amounts. In the final spray-dried granule, the hydratable material is preferably hydrated to an extent of from 10% to 90%, preferably from 30% to 60% of its hydration capacity (based on the highest hydrate which is stable at room temperature). Preferably, the inorganic hydratable material has no hydrate transition point below 40°C or consists majorly of such material (greater than 50%, preferably greater than 75% of total inorganic hydratable material).

In the present invention, pH control is all-important both for successful spray drying of the aqueous

slurry mix and for stability of the resulting granular composition. In particular, the provision of a pH-regulated outer coating based on partly-hydrated inorganic hydratable material appears to be especially beneficial in providing both physical and chemical stabilization of the water-soluble bleach activator in the final detergent or bleach product. In the practice of the invention the pH of the granular composition (1% aqueous solution) should lie within the range from 3 to 8, preferably from 3.5 to 7, more preferably from 4 to 6.5, while the aqueous slurry for spray-drying should also be adjusted to within the same pH range (1% solids solution). Adjustment of pH can be achieved in various ways, for example, by incorporating inorganic acid salts having the required pH characteristics, or by adding mineral acids or organic acids to the crutcher mix to lower the pH into the prescribed range. Suitable organic acids herein include C₈—C₂₄ fatty acids, polycarboxylic acids, aminopolycarboxylic acids, polyphosphonic acids, aminopolyphosphonic acids and mixtures thereof.

The spray-dried granular bleach activator compositions herein generally comprise from 0.1% to 50%, preferably from 1% to 45% bleach precursor, and from 10% to 99.9%, preferably from 25% to 90%, more preferably from 35% to 90% alkaline inorganic hydratable material. In addition, they generally have a bulk density of from 300 to 900 grams/litre, preferably from 450 to 750 grams/litre, and a weight average particle size of from 0.15 to 3 mm, preferably from 0.5 mm to 1.4 mm.

The aqueous slurry for spray drying comprises generally from 30% to 60% water, from 0.05% to 35%, preferably from 0.5% to 30%, more preferably from 2% to 25% bleach precursor, and from 10% to 69.95%, preferably from 14% to 63% alkaline inorganic hydratable material. The slurry is then heated to a temperature of from 60°C to 90°C and spray-dried in a current of air having an inlet temperature of from 250°C to 350°C, preferably from 275°C to 330°C, and an outlet temperature of from 95°C to 125°C, preferably from 100°C to 115°C.

The bleach activators used in the compositions and process of the invention are water-soluble materials, being soluble generally to an extent of at least 1%, preferably at least 5% by weight at 25°C and pH 7. By 'soluble' is meant that the bleach activator/water system is free of solids at the specified concentration. The preferred bleach activator herein is a peroxyacid bleach precursor having the general formula!

wherein R is an optionally substituted alkyl group containing from 6 to 12 carbon atoms wherein the longest linear alkyl chain extending from and including the carbonyl carbon contains from 5 to 10 carbon atoms and L is a leaving group containing an anionic molety, the conjugate acid of the leaving group having a pK_a in the range from 6 to 13.

The group R can be either linear or branched and, in preferred embodiments, it is unsubstituted and contains from 7 to 9 carbon atoms. In another group of suitable bleach activators, the group R is substituted and has the general formula II

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wherein R¹ is straight or branched chain alkyl containing from 4 to 10, preferably 6 to 10, more preferably 6 to 8 carbon atoms, R^2 is H, CH_3 , C_2H_8 or C_3H_7 and X is Cl, Br, OCH_3 or OC_2H_8 .

L can be essentially any suitable leaving group containing a moiety which is anionic at pH 7. A leaving group is any group that is displaced from the bleach activator as a consequence of the nucleophilic attack on the bleach activator by the perhydroxide anion. Generally, for a group to be a suitable leaving group it must exert an electron attracting effect. Leaving groups that exhibit such behavior are those in which their conjugate acid has a pK_a in the range of from 6 to 13, preferably from 7 to 11 and most preferably from 8 to 11. Also, in order for the activator to have the desired level of solubility in wash water it is essential that the leaving group contain an anionic moiety. Nonlimiting examples of suitable anionic moieties are —SO₃M, —COOM and —OSO₃M wherein M is a proton or a compatible cation.

Preferred bleech activators are those of the general formula I wherein L is selected from

and

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a)

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wherein Z is H, R³ or halogen, R³ is an alkyl group having from 1 to 4 carbon atoms, x is 0 or an integer of from 1 to 4 and Y is selected from SO₃M, OSO₃M and CO₂M and wherein M is H, alkali metal, alkaline earth metal, ammonium or substituted ammonium.

The preferred leaving group L has the formula (a) in which Z is H, x is O and Y is sulfonate or carboxylate. Highly preferred materials are sodium 3,5,5-trimethylhexanoyloxybenzene sulfonate, sodium 3,5,5-trimethylhexanoyloxybenzenesulfonate, sodium nonanoyloxybenzenesulfonate, sodium 2-ethlorooctanoyloxybenzenesulfonate, sodium 2-chlorooctanoyloxybenzenesulfonate, sodium 2-chlorooctanoyloxybenzenesulfonate, sodium 3,5,5-trimethyl-2-chlorohexanoyloxybenzenesulfonate, and sodium 2-chloro-2-ethyl-hexanoyloxybenzenesulfonate, the acyloxy group in each instance preferably being p-substituted.

The present invention also encompasses bleaching compositions, laundry detergent and laundry additive compositions comprising the bleach activator compositions detailed herein as well as laundry compositions incorporating the water-soluble bleach activator and detergent in a co-dried granule. Bleaching compositions according to the invention suitably contain from 5% to 99.5%, preferably from 20% to 90% of peroxygen bleaching agent and from 0.5% to 95%, preferably from 10% to 80% of bleach activator composition. Laundry compositions according to the invention generally contain from 2% to 40%, preferably from 5% to 25% of detersive surfactant selected from anionic, nonionic, cationic, ampholytic and zwitterionic surfactants and mixtures thereof and from 0.1% to 20%, preferably from 0.5% to 10% of the water-soluble organic peroxyacid bleach precursor. The surfactant and bleach precursor can either be co-spray dried or the surfactant can be incorporated in a separate laundry base composition. When co-spray dried, the spray dried granules contain the water-soluble or water-dispersible inorganic hydratable material at a level of from 10% to 90%, preferably from 15% to 70% by weight of the laundry composition and the spray-dried granules generally constitute from 15% to 100%, preferably from 25% to 75% by weight of the laundry composition. When the surfactant is incorporated in a separate laundry base component, the bleach activator composition generally comprises from 0.5% to 40%, preferably from 1% to 10% by weight of the laundry composition, and the base composition comprises from 25% to 99.5%, preferably from 35% to 75% by weight of the laundry composition. In addition, the laundry compositions generally comprise one or more inorganic or organic detergency builders in a total level of from 15% to 90%, preferably from 20% to 60% by weight of the laundry composition, and peroxygen bleaching agent in a level of from 5% to 35%, preferably from 8% to 20% by weight of the laundry composition.

A wide range of surfactants can be used in the laundry compositions of the invention. US—A—4,111,855 and US—A—3,995,669 contain detailed listing of typical detersive surfactants.

Suitable synthetic anionic surfactants are water-soluble salts of C_8 — C_{22} alkyl benzene sulphonates, C_8 — C_{22} alkyl sulphates, C_{10-18} alkyl polyethoxy ether sulphates, C_{8-24} paraffin sulphanates, alpha- C_{12-24} olefin sulphonates, alpha-sulphonated C_8 — C_{20} fatty acids and their esters, C_{10} — C_{18} alkyl glyceryl ether sulphonates, fatty acid monoglyceride sulphates and sulphonates, especially those prepared from coconut oil, C_8 — C_{12} alkyl phenol polyethoxy ether sulphates, 2-acyloxy C_8 — C_{23} alkane-1-sulphonates, and beta-alkyloxy C_8 — C_{20} alkane sulphonates.

A particularly suitable class of anionic surfactants includes water-soluble salts, particularly the alkali metal, ammonium and alkanolammonium salts or organic sulphuric reaction products having in their molecular structure an alkyl or alkaryl group containing from 8 to 22, especially from 10 to 20 carbon atoms and a sulphonic acid or sulphuric acid ester group. (Included in the term "alkyl" is the alkyl portion of acyl groups).

Examples of this group of synthetic detergents are the sodium and potassium alkyl sulphates, especially those obtained by sulphating the higher alcohols (C_{8-19}) carbon atoms produced by reducing the glycerides of tallow or coconut oil and sodium and potassium alkyl benzene sulphonates, in which the alkyl group contains from 9 to 15, especially 11 to 13, carbon atoms, in straight chain or branched chain configuration, e.g. those of the type described in US—A—2,220,099 and US—A—2,477,383 and those prepared from alkylbenzenes obtained by alkylation with straight chain chloroparaffins (using aluminium trichloride catalysis) or straight chain olefins (using hydrogen fluoride catalysis). Especially valuable are linear straight chain alkyl benzene sulphonates in which the average of the alkyl group is about 11.8 carbon atoms, abbreviated as $C_{11.8}$ LAS, and C_{12} — C_{15} methyl branched alkyl sulphates.

The alkane chains of the foregoing non-soap anionic surfactants can be derived from natural sources such as coconut oil or tallow, or can be made synthetically as for example using the Ziegler or Oxo processes. Water solubility can be achieved by using alkali metal, ammonium or alkanolammonium cations; sodium is preferred.

Suitable fatty acid soaps herein can be selected from the ordinary alkali metal (sodium, potassium), ammonium, and alkylolammonium salts of higher fatty acids containing from 8 to 24, preferably from 10 to 22 and especially from 16 to 22 carbon atoms in the alkyl chain. Fatty acids in partially neutralized form are

also suitable for use herein, especially in liquid compositions. Sodium and potassium soaps can be made by direct saponification of the fats and oils or by the neutralization of the free fatty acids which are prepared in a separate manufacturing process. Particularly useful are the sodium and potassium salts of the mixtures of fatty acids derived from tallow and hydrogenated fish oil.

Mixtures of anionic surfactants are particularly suitable herein, especially mixtures of sulphonate and sulphate surfactants in a weight ratio of from 5:1 to 1:5, preferably from 5:1 to 1:1, more preferably from 5:1 to 1.5:1. Especially preferred is a mixture of an alkyl benzene sulphonate having from 9 to 15, especially 11 to 13 carbon atoms in the alkyl radical, the cation being an alkali metal, preferably sodium; and either an alkyl sulphate having from 10 to 20, preferably 12 to 18 carbon atoms in the alkyl radical or an ethoxy sulphate having from 10 to 20, preferably 10 to 16 carbon atoms in the alkyl radical and an average degree of ethoxylation of 1 to 6, having an alkali metal cation, preferably sodium.

The nonionic surfactants useful in the present invention are condensates of ethylene oxide with a hydrophobic moiety to provide a surfactant having an average hydrophilic-lipophilic balance (HLB) in the

range from 8 to 17, preferably from 9.5 to 13.5, more preferably from 10 to 12.5.

Examples of suitable nonionic surfactants include the condensation products of primary or secondary aliphatic alcohols having from 8 to 24 carbon atoms, in either straight chain or branched chain configuration, with from 2 to 40 moles, preferably 2 to 9 moles of ethylene oxide per mole of alcohol. Preferably, the cliphatic alcohol comprises between 9 and 18 carbon atoms and is ethoxylated with between 2 and 9, desirably between 3 and 8 moles of ethylene oxide per mole of aliphatic alcohol. The preferred surfactants are prepared from primary alcohols which are either linear (such as those derived from natural fats or, prepared by the Ziegler process from ethylene, e.g. myristyl, cetyl, stearyl alcohols), or partly branched such as the Lutensols (RTM), Dobanols (RTM) and Neodols (RTM) which have about 25% 2-methyl branching (Lutensol (RTM) being a Trade Name of BASF, Dobanol (RTM) and Neodol (RTM) being Trade Names of Shell), or Synperonics (RTM), which are understood to have about 50% 2-methyl branching (Synperonic (RTM) is a Trade Name of I.C.I.) or the primary alcohols having more than 50% branched chain structure sold under the Trade Name Lial by Liquichimica. Specific examples of nonionic surfactants falling within the scope of the invention include Dobanol (RTM) 45-4, Dobanol (RTM) 45-7, Dobanol (RTM) 45-9, Dobanol (RTM) 91-2.5, Dobanol (RTM) 91-3, Dobanol (RTM) 91-4, Dobanol (RTM) 91-6, Dobanol (RTM) 91-8, Dobanol (RTM) 23-6.5, Synperonic (RTM) 6, Synperonic (RTM) 14, the condensation products of coconut alcohol with an average of between 5 and 12 moles of ethylene oxide per mole of alcohol, the coconut alkyl portion having from 10 to 14 carbon atoms, and the condensation products of tallow alcohol with an average of between 7 and 12 moles of ethylene oxide per mole of alcohol, the tallow portion comprising essentially between 16 and 22 carbon atoms. Secondary linear alkyl ethoxylates are also suitable in the present compositions, especially those ethoxylates of the Tergitol series having from 9 to 15 carbon atoms in the alkyl group and up to 11, especially from 3 to 9, ethoxy residues per molecule.

Other suitable nonionic surfactants include the condensation products of C₈—C₁₂ alkyl phenols with from 3 to 30, preferably 5 to 14 moles of ethylene oxide, and the compounds formed by condensing ethylene oxide with a hydrophobic base formed by the condensation of propylene oxide with propylene glycol, such synthetic nonionic detergents being available on the market under the Trade Name of "Pluronic (RTM)" supplied by Wyandotte Chemicals Corporation.

Especially preferred nonionic surfactants for use herein are the C₅—C₁₅ primary alcohol ethoxylates containing 3—8 moles of ethylene oxide per mole of alcohol, particularly the C₁₂—C₁₅ primary alcohols

containing 6—8 moles of ethylene oxide per mole of alcohol.

Cationic surfactants suitable for use herein include quaternary ammonium surfactants and surfactants of a semi-polar nature, for example amine oxides. Suitable quaternary ammonium surfactants are selected from mono C_8 — C_{16} , preferably C_{10} — C_{14} N-alkyl or alkenyl ammonium surfactants wherein remaining N positions are substituted by methyl, hydroxyethyl or hydroxypropyl and the corresponding di- C_8 — C_{10} N-alkyl or alkenyl ammonium surfactants. Suitable amine oxides are selected from mono C_8 — C_{20} , preferably C_{10} — C_{14} N-alkyl or alkenyl amine oxides and propylene-1,3-diamine dioxides wherein the remaining N positions are again substituted by methyl, hydroxyethyl or hydroxypropyl.

Suitable detergent builder salts useful herein can be of the polyvalent inorganic and polyvalent organic types, or mixtures thereof. Non-limiting examples of suitable water-soluble, inorganic alkaline detergent builder salts include the alkali metal carbonates, borates, phosphates, pyrophosphates, tripolyphosphates

and bicarbonates.

Organic builder/chelating agents that can be incorporated include citric acid, nitrilotriacetic and ethylenediamine tetra acetic acids and their salts, organic phosphonate derivatives such as those disclosed in US—A—3,213,030, US—A—3,433,021, US—A—3,292,121 and US—A—2,599,807, and carboxylic acid builder salts such as those disclosed in US—A—3,308,067. Preferred chelating agents include nitrilotriacetic acid (NTA), nitrilo(trimethylene phosphonic acid) (NTMP), ethylenediamine tetra(methylene phosphonic acid) (EDTMP) and diethylenetriamine penta(methylene phosphonic acid) (DETPMP). Mixtures of organic and/or inorganic builders can be used herein. One such mixture of builders is disclosed in CA—A—755,038, e.g. a ternary mixture of sodium tripolyphosphate, trisodium nitrilotriacetate, and trisodium ethane-1-hydroxy-1,1-diphosphonate.

A further class of builder salts is the insoluble alumino silicate type which functions by cation exchange

to remove polyvalent mineral hardness and heavy metal ions from solution. A preferred builder of this type has the formulation $Na_z(AlO_z)_z(SiO_z)_y$. xH_2O wherein z and y are integers of at least 6, the molar ratio of z to y is in the range from 1.0 to 0.5 and x is an integer from 15 to 264. Compositions incorporating builder salts of this type form the subject of GB—A—1,429,143, DE—A—2,433,485, and DE—A—2,525,778.

An alkali metal, or alkaline earth metal, silicate can also be present in granular compositions of the invention. The alkali metal silicate is preferably from 3% to 15%. Suitable silicate solids have a molar ratio

of SiO₂/alkali metal₂O in the range from 1.0 to 3.3, more preferably from 1.5 to 2.0.

The compositions herein can also contain bleaching components. In general, the bleach is selected from inorganic peroxy salts, hydrogen peroxide, hydrogen peroxide adducts, and organic peroxy acids and salts thereof. Suitable inorganic peroxygen bleaches include sodium perborate mono- and tetrahydrate, sodium percarbonate, sodium persilicate, urea-hydrogen peroxide addition products and the clathrate $4Na_2SO_4:2H_2O_2:1NaCl$. Suitable organic bleaches include peroxylauric acid, peroxyoctanoic acid, peroxynonanoic acid, peroxydecanoic acid, diperoxydecanoic acid, diperoxyazelaic acid, mono- and diperoxyphthalic acid and mono- and diperoxyisophthalic acid.

The compositions of the invention can be supplemented by all manner of detergent and laundering components, inclusive of suds suppressors, enzymes, fluorescers, photoactivators, soil suspending agents,

anti-caking agents, pigments, perfumes, fabric conditioning agents etc.

Suds suppressors are represented by materials of the silicone, wax, vegetable and hydrocarbon oil and phosphate ester varieties. Suitable silicone suds controlling agents include polydimethylsiloxanes having a molecular weight in the range from 200 to 200,000 and a kinematic viscosity in the range from 20 to 2,000,000 mm²/s, preferably from 3000 to 30,000 mm²/s, and mixtures of siloxanes and hydrophobic silanated (preferably trimethylsilanated) silica having a particle size in the range from about 10 nm to 20 nm and a specific surface area above 50 m²/g. Suitable waxes include microcrystalline waxes having a melting point in the range from 65°C to 100°C, a molecular weight in the range from 4000—1000, and a penetration value of at least 6, measured at 77°C by ASTM—D1321, and also paraffin waxes, synthetic waxes and natural waxes. Suitable phosphate esters include mono- and/or di-C₁₆—C₂₂ alkyl or alkenyl phosphate esters, and the corresponding mono- and/or di-alkyl or alkenyl ether phosphates containing up to 6 ethoxy groups per molecule.

Enzymes suitable for use herein include those discussed in US—A—3,519,570 and US—A—3,533,139. Suitable fluorescers include Blankophor (RTM) MBBH (Bayer AG) and Tinopal (RTM) CBS and EMS (Ciba Geigy). Photoactivators are discussed in EP—A—57088, highly preferred materials being zinc phthalocyanine, tri- and tetra-suifonates. Suitable fabric conditioning agents include smectite-type clays as

disclosed in GB-A-1400898 and di-C₁₂-C₂₄ alkyl or alkenyl amines and ammonium salts.

Antiredeposition and soil suspension agents suitable herein include cellulose derivatives such as methylcellulose, carboxymethylcellulose and hydroxyethylcellulose, and homo- or co-polymeric polycarboxylic acids or their salts in which the polycarboxylic acid comprises at least two carboxyl radicals separated from each other by not more than two carbon atoms. Polymers of this type are disclosed in GB—A—1,596,756. Preferred polymers include copolymers or salts thereof of maleic anhydride with ethylene, methylvinyl ether, acrylic acid or methacrylic acid, the maleic anhydride constituting at least 20 mole percent of the copolymer. These polymers are valuable for improving whiteness maintenance, fabric ash deposition, and cleaning performance on clay, proteinaceous and oxidizable soils in the presence of transition metal impurities.

Laundry additive products comprising bleaching or laundry detergent compositions in water-releasable combination with a non-particulate carrier as described in EP—A—96566 and EP—A—99197, are also suitable herein. A preferred carrier takes the form of a flexible sheet-like substrate, for example, an apertured non-woven fabric made of rayon with a nonionic self-crosslinking acrylic polymer as binder, the weight ratio of bleaching or bleaching detergent composition to substrate being at least 1:1, preferably at least 3:1, more preferably at least 6:1. Carriers in the form of laminates, pouches and the like are also suitable, however.

In the Examples which follow, the abbreviations used have the following designations:-

LAS Linear C_{11.8} alkyl benzene sulphonate. TAE(n) Hardened tallow alcohol ethoxylated with n moles of ethylene oxide. C₁₂₋₁₄ alcohol sulfate, sodium salt. AS TAS Tallow alcohol sulfate. **CATAB** Coconut alkyl trimethyl ammonium bromide Dobanol (RTM) 45-A C14-15 oxo-alcohol with 7 moles of ethylene oxide, marketed by Shell. Silicate Sodium silicate having an SiO2:Na2O ratio of 1.6:1. NTA Sodium nitrilotriacetate. Gantrez (RTM) AN119 Trade Name for maleic anhydride/vinyl methyl ether co-polymer, believed to have an average molecular weight of about 240,000, marketed by GAF. This was prehydrolysed with NaOH before addition.

: Copolymer of 1:4 maleic acid/acrylic acid, average molecular weight about

80.000.

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MA/AA

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	Brightener .	:	Disodium 4,4'-bis(2-morpholino-4-anilino-s-triazino-6-ylamino)stilbene- 2:2'-disulphonate.
	Dequest (RTM) 2060	:	Trade Name for diethylenetriaminepenta(methylenephosphonic acid), marketed by Monsanto.
5	Dequest (RTM) 2041	:	Trade Name for ethylenediamine tetra(methylene phosphonic acid)monohydrate, marketed by Monsanto.
	INOBS	:	Sodium 3,5,5-trimethyl hexanoyl oxybenzene sulphonate.
	CNOBS	:	Sodium 4-(2-chlorononanoyloxy) benzene sulphonate.
	Perborate		Sodium perborate tetrahydrate.
10	Clay	:	Sodium montmorillonite.
	Zeolite A	:	0.4 nm (4 Å) pore size.
	Enzyme	:	Protease.

The present invention is illustrated by the following non-limiting examples:--

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Examples I to VI

Granular detergent compositions are prepared as follows. A detergent base powder composition is first prepared by mixing the indicated components in a crutcher as an equeous slurry at a temperature of 70°C and containing 35% water. The slurry is then spray dried at a gas inlet temperature of 300°C to form base powder granules. In Examples I to III, the detergent base powder contains water-soluble bleach activator in addition to the detersive components. In Examples IV to VI, a separate bleach activator composition is prepared by mixing the indicated components in a crutcher at a temperature of 70°C and containing 38% water, the slurry being spray dried at a gas inlet temperature of 300°C and a gas outlet temperature of 100°C. Finally, the detergent base powder composition is dry mixed with enzyme, silicate, carbonate, bleach and spray-dried bleach activator components, where present, and additional nonlonic surfactant acid, where present, is sprayed onto the total mixture.

All percentages are given by weight of total detergent composition.

EXAMPLES

30	ur.	ı	11	101	IV	٧	VI	
	 Detergent Base Powder							
35	LAS	6	5	8	5	8	5	
	AS	4		3	_	_	5	
40	TAS	<u> </u>	5	_	5	1	5	
	CATAB	_	_	2		1	_	
45	Gantrez (RTM) AN119	_				1	_	
	Silicate	-	<u> </u>	_	_. 5	1	3	
	MA/AA	0.5	. 2	1	2	_	3	
50	Zeolite A	18 .		_		- .		
	Brightener	0.2	0.3	0.1	0.2	0.2	0.3	
	NTA	3	.		_	_ `	-	
55	Dequest (RTM) 2060		_	0.3		· -	_	
60	Dequest (RTM) 2041	0.1	0.3	_	0.3	0.1	- .	
	EDTA	0.2	0.3		0.2	0.2	0.2	
	INOBS	5	2.5	- .	_	_		
	Sodium tripolyphosphate		24	28	25	30	22	
65	CNOBS	_	_	2	· _	_	_	

EXAMPLES (Continued)

		1	, II	. 101	IV	٧	VI	
5	Magnesium sulphate (ppm)	1000	1000	800	1000	1200	_	
10	Sodium sulphate, moisture and miscellaneous		to 100					
	pH (1% solution)	6.8	7.5	6.5	10.1	9.8	9.9	
15	Bleach Activator Composition							
	INOBS	_	·	_	4	-	2	
20	CNOBS		· 		_	4	_	
	Sodium tripolyphosphate	_	_	· —	. 3	3	1	
	Disodium dihydrogen pyrophosphate	_	- .	· —	1	2	0.5	
25	Sodium sulphate	_	· — ·		0.5	1	0.5	
	Dequest (RTM) 2041		_	_		_	0.5	
30	Water	-	_	_	1	1	0.5	
	pH (1% solution)	_	_		6.5	6.2	6.5	
	OTHER ADDITIVES				•			
35	Enzyme	0.4	0.6	1.0	0.5	0.6	_	
	Sodium perborate tetrahydrate	24	20	14	21	22	-	
40	Dobanol (RTM) 45—E—7	5	4	_	2	4		
	Silicate	2	5	7	_	_	_	
	Sodium carbonate	18	13		10	_	– .	

The above products combine excellent bleach activator stability, water-dispersibility, granulometry and detergency performance across the range of wash temperatures and soil types.

Claims

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1. A bleach activator composition in the form of spray-dried granules having an inner core comprising organic peroxyacid bleach precursor and a surface coating comprising a water-soluble or water-dispersible, inorganic hydratable material, characterised in that the organic peroxyacid bleach precursor is water-soluble and wherein the composition has a pH (1% aqueous solution) in the range from 3 to 8.

2. A composition according to Claim 1 wherein the inner core comprises an essentially spherical

droplet of peroxyacid bleach precursor.

3. A composition according to Claim 1 or 2 wherein the organic peroxy bleach precursor has the general formula I

₽—C—

wherein R is an optionally substituted alkyl group containing from 6 to 12 carbon atoms wherein the longest linear alkyl chain extending from and including the carbonyl carbon contains from 5 to 10 carbon

atoms and L is a leaving group containing an anionic moiety, the conjugate acid of the leaving group having a pK_a in the range from 6 to 13.

4. A composition according to any of Claims 1 to 3 wherein the inorganic hydratable material consists majorly of one or more acid salts having a pKa in the range from 1 to 9, preferably from 2 to 7, more preferably from 3 to 6.5.

5. A composition according to any of Claims 1 to 4 having a pH in the range from 3.5 to 7, preferably from 4 to 6.5.

6. A composition according to any of Claims 1 to 5 wherein the inorganic hydratable material is selected from disodium dihydrogen pyrophosphate, tetrasodium monohydrogen tripolyphosphate, trisodium dihydrogen tripolyphosphate, sodium sulphate, magnesium sulphate, sodium bicarbonate, acidified pentasodium tripolyphosphate, acidified borax and acidified aluminosilicates and mixtures

7. A composition according to any of Claims 1 to 6 additionally comprising an organic acid selected from C₈—C₂₄ fatty acids, polycarboxylic acids, aminopolycarboxylic acids, polyphosphonic acids, aminopolyphosphonic acids and mixtures thereof.

8. A composition according to any of Claims 1 to 7 wherein the inorganic hydratable material consists

majorly of one or more materials having no transition point below 40°C.

9. A composition according to any of Claims 1 to 8 comprising from 0.1% to 50%, preferably from 1% to 45% by weight of bleach precursor, and from 25% to 99.9%, preferably from 35% to 90% by weight of inorganic hydratable material.

10. A composition according to any of Claims 1 to 9 having a bulk density of from 300 to 900 grams/litre, preferably from 400 to 750 grams per litre and a weight average particle size of from 0.15 to 3 mm, preferably from 0.5 mm to 0.14 mm.

11. A bleach activator composition according to Claim 1 comprising from 0.1% to 50% by weight of the water-soluble organic peroxyacid bleach precursor and from 10% to 99.9% by weight of the water-soluble or water-dispersible inorganic hydratable material, the inorganic hydratable material consisting majorly of one or more acid salts having a pK_a in the range from 1 to 9.

12. A process of making a granular bleach activator composition according to Claim 1 comprising forming an aqueous slurry of the water-soluble organic peroxyacid bleach precursor and the water-soluble or water-dispersable inorganic hydratable material, adjusting the pH of the slurry (1% solids) to within the

range from 3 to 8, preferably from 3.5 to 7 and subjecting the slurry to spray-drying.

13. A process according to Claim 12 wherein the slurry comprises from 30% to 60% by weight of water, from 0.05% to 35%, preferably from 0.5% to 30%, more preferably from 2% to 25% by weight of bleach precursor, and from 10% to 69.95%, preferably from 14% to 63% by weight of inorganic hydratable

14. A process according to Claim 12 or 13 wherein the slurry has a temperature of from 60°C to 90°C and is spray dried at an air-inlet temperature of from 250°C to 350°C and an air-outlet temperature of from 95°C to 115°C.

15. A bleaching composition comprising from 5% to 99.5% by weight of peroxygen bleaching agent characterised in that the bleaching composition additionally comprises from 0.5% to 95% by weight of a bleach activator composition in the form of spray-dried granules according to any of Claims 1 to 11.

16. A laundry composition comprising from 25% to 99.5% by weight of a laundry base composition comprising from 2% to 40% by weight of the laundry composition of surfactant selected from anionic, nonionic, cationic, ampholytic and zwitterionic surfactants and mixtures thereof, characterised in that the laundry composition additionally comprises from 0.5% to 40% by weight of a bleach activator composition in the form of spray-dried granules according to any of Claims 1 to 11.

17. A laundry composition in the form of spray-dried granules comprising by weight of the composition from 0.1% to 20% of organic peroxyacid bleach precursor, from 2% to 40% of surfactant selected from anionic, nonionic, cationic, ampholytic and zwitterionic surfactants and mixtures thereof, and from 10% to 90% of water-soluble or water-dispersible inorganic hydratable material consisting majorly of one or more acid salts having a pKa in the range from 1 to 9, characterised in that the organic peroxyacid bleach precursor is water-soluble and wherein the granules have a pH (1% aqueous solution) in the range from 3 to 8.

18. A composition according to Claim 16 comprising from 15% to 90% by weight of inorganic or organic detergency builder and from 5% to 35% by weight of peroxygen bleaching agent.

Patentansprüche

1. Eine Bleichmittelaktivatorzusammensetzung in der Form von sprühgetrockneten Körnern, welche einen organischen Peroxysäurebleichmittelprecursor enthaltenden Innenkern und einen ein wasserlösliches oder wasserdispergierbares, anorganisches, hydratisierbares Material enthaltenden Oberflächenüberzug aufweisen, dadurch gekennzeichnet, daß der organische Peroxysäurebleichmittelprecursor wasserlöslich ist, und daß die Zusammensetzung einen pH-Wert (1%ige wässerige Lösung) im Bereich von 3 bis 8 hat.

- Eine Zusammensetzung nach Anspruch 1, wobei der Innenkern ein im wesentlichen kugelförmiges Tröpfchen von Peroxysäurebleichmittelprecursor umfaßt.
- 3. Eine Zusammensetzung nach Anspruch 1 oder 2, wobei der organische Peroxybieichmittelprecursor die allgemeine Formel I

O || |R---C---|

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hat, worin R eine gegebenenfalls substituierte, 6 bis 12 Kohlenstoffatome enthaltende Alkylgruppe ist, wobei die längste gerade Alkylkette, die sich vom Carbonylkohlenstoff erstreckt und diesen umfaßt, 5 bis 10 Kohlenstoffatome enthält, und L eine austretende Gruppe ist, die einen anionischen Rest enthält, wobei die Konjugatsäure der austretenden Gruppe einen pK₀-Wert im Bereich von 6 bis 13 hat.

4. Eine Zusammensetzung nach einem der Ansprüche 1 bis 3, wobei das anorganische, hydratisierbare Material mehrheitlich aus einem oder mehreren Säuresalzen mit einem pK_α-Wert im Bereich von 1 bis 9, vorzugsweise von 2 bis 7, und in höher bevorzugtem Maße von 3 bis 6,5, besteht.

5. Eine Zusammensetzung nach einem der Ansprüche 1 bis 4, die einen pH-Wert im Bereich von 3,5 bis 7, vorzugsweise von 4 bis 6,5, aufweist.

6. Eine Zusammensetzung nach einem der Ansprüche 1 bis 5, wobei das anorganische, hydratisierbare Material aus Dinatriumdihydrogenpyrophosphat, Tetranatriummonohydrogentripolyphosphat, Trinatriumdihydrogentripolyphosphat, Natriumsulfat, Magnesiumsulfat, Natriumbicarbonat, angesäuertem Pentanatriumtripolyphosphat, angesäuertem Borax und angesäuerten Aluminosilicaten, und Gemischen davon, ausgewählt ist.

7. Eine Zusammensetzung nach einem der Ansprüche 1 bis 6, welche zusätzlich eine organische Säure enthält, die aus C₈—C₂₄-Fettsäuren, Polycarbonsäuren, Aminopolycarbonsäuren, Polyphosphonsäuren, Aminopolyphosphonsäuren, und Gemischen davon, ausgewählt ist.

8. Eine Zusammensetzung nach einem der Ansprüche 1 bis 7, wobei das anorganische, hydratisierbare Material mehrheitlich aus einem oder mehreren Materialien besteht, die keinen Phasenübergangspunkt unter 40°C aufweisen.

9. Eine Zusammensetzung nach einem der Ansprüche 1 bis 8, enthaltend 0,1 Gew.-% bis 50 Gew.-%, vorzugsweise 1 Gew.-% bis 45 Gew.-%, an Bleichmittelprecursor, und 25 Gew.-% bis 99,9 Gew.-%, vorzugsweise 35 Gew-% bis 90 Gew.-%, an anorganischem, hydratisierbarem Material.

10. Eine Zusammensetzung nach einem der Ansprüche 1 bis 9, die ein Schüttgewicht von 300 bis 900 g/l, vorzugsweise von 400 bis 750 g/l, und eine Teilchengröße im gewogenen Durchschnitt von 0,15 bis 3 mm, vorzugsweise von 0,5 mm bis 0,14 mm, aufweist.

11. Eine Bleichmittelaktivatorzusammensetzung nach Anspruch 1, enthaltend 0,1 Gew.-% bis 50 Gew.-% des wasserlöslichen, organischen Peroxysäurebleichmittelprecursors und von 10 Gew.-% bis 99,9 Gew.-% des wasserlöslichen oder wasserdispergierbaren, anorganischen, hydratisierbaren Materials, wobei das anorganische, hydratisierbare Material mehrheitlich aus einem oder mehreren Säuresalzen mit einem pKa-Wert im Bereich von 1 bis 9 besteht.

12. Ein Verfahren zur Herstellung einer k\u00fcrnigen Bleichmittelaktivatorzusammensetzung nach Anspruch 1, umfassend das Bilden einer w\u00e4sserigen Aufschl\u00e4mmung des wasserl\u00f6slichen, organischen Peroxys\u00e4urebleichmittelprecursors und des wasserl\u00f6slichen oder wasserdispergierbaren, anorganischen, hydratisierbaren Materials, das Einstellen des pH-Wertes der Aufschl\u00e4mmung (1% Feststoffe) auf einen Wert im Bereich von 3 bis 8, vorzugsweise von 3,5 bis 7, und das Unterwerfen der Aufschl\u00e4mmung dem Spr\u00fchtrocknen,

13. Ein Verfahren nach Anspruch 12, wobei die Aufschlämmung 30 Gew.-% bis 60 Gew.-% Wasser, 0,05 Gew.-% bis 35 Gew.-%, vorzugsweise 0,5 Gew.-% bis 30 Gew.-%, und in höher bevorzugtem Maße 2 Gew.-% bis 25 Gew.-%, Bleichmittelprecursor, und 10 Gew.-% bis 69,95 Gew.-%, vorzugsweise 14 Gew.-% bis 63 Gew.-%, anorganisches, hydratisierbares Material enthält.

14. Ein Verfahren nach Anspruch 12 oder 13, wobei die Aufschlämmung eine Temperatur von 60°C bis 90°C hat und bei einer Lufteinlaßtemperatur von 250°C bis 350°C und einer Luftauslaßtemperatur von 95°C bis 115°C sprühgetrocknet wird.

15. Eine Bleichmittelzusammensetzung, enthaltend 5 Gew.-% bis 99,5 Gew.-% eines Persauerstoffbleichmittels, dadurch gekennzeichnet, daß die Bleichmittelzusammensetzung zusätzlich 0,5 Gew.-% bis 95 Gew.-% einer Bleichmittelaktivatorzusammensetzung in der Form von sprühgetrockneten Körnern nach einem der Ansprüche 1 bis 11 enthält.

16. Eine Wäschewaschmittelzusammensetzung, enthaltend 25 Gew.-% bis 99,5 Gew.-% einer Wäschewaschmittelbasiszusammensetzung mit einem Gehalt von 2 Gew.-% bis 40 Gew.-% der Wäschewaschmittelzusammensetzung an grenzflächenaktivem Mittel, ausgewählt aus anionischen, nichtlonischen, kationischen, ampholytischen und zwitterlonischen grenzflächenaktiven Mitteln, und Gemischen davon, dadurch gekennzeichnet, daß die Wäschewaschmittelzusammensetzung zusätzlich 0,5 Gew.-% bis 40 Gew.-% einer Bleichmittelaktivatorzusammensetzung in der Form von sprühgetrockneten Körnern nach einem der Ansprüche 1 bis 11 enthält.

17. Eine Wäschewaschmittelzusammensetzung in der Form von sprühgetrockneten Körnern, enthaltend, bezogen auf das Gewicht der Zusammensetzung, 0,1% bis 20% an organischem Peroxysäurebleichmittelprecursor, 2% bis 40% grenzflächenaktives Mittel, ausgewählt aus anionischen, nichtlonischen, kationischen, ampholytischen und zwitterionischen grenzflächenaktiven Mitteln, und Gemischen davon, und 10% bis 90% an wasserlöslichem oder wasserdisperglerbarem, anorganischem, hydratisierbarem Material, das mehrheitlich aus einem oder mehreren Säuresalzen mit einem pKa-Wert im Bereich von 1 bis 9 besteht, dadurch gekennzeichnet, daß der organische Peroxysäurebleichmittelpercursor wasserlöslich ist, und daß die Körner einen pH-Wert (1%ige wässerige Lösung) im Bereich von 3 bis 8 haben.

18. Eine Zusammensetzung nach Anspruch 16, enthaltend 15 Gew.-% bis 90 Gew.-% anorganischen oder organischen Detergensgerüststoff und 5 Gew.-% bis 35 Gew.-% Persauerstoffbleichmittel.

Revendications

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1. Composition d'activateur de blanchiment se présentant sous forme de granules séchés par atomisation comportant une partie interne comprenant un précurseur d'agent de blanchiment de type peracide organique et un enrobage de surface comprenant une substance minérale hydratable, celuble dans l'eau ou dispersable dans l'eau, caractérisée en ce que le précurseur d'agent de blanchiment de type peracide organique est soluble dans l'eau et en ce que la composition a un pH (en solution aqueuse à 1%) dans l'intervalle de 3 à 8.

Composition selon la revendication 1, dans laquelle la partie interne comprend une gouttelette, essentiellement sphérique, de précurseur d'agent de blanchiment de type peracide.

3. Composition selon la revendication 1 ou 2, dans laquelle le précurseur d'agent de blanchiment peroxygéné organique répond à la formule générale l



dans laquelle R est un groupe alkyle éventuellement substitué, contenant de 6 à 12 atomes de carbone, dans lequel la chaîne alkyle linéaire la plus longue partant de l'atome de carbone du groupement carbonyle et incluant cet atome contient de 5 à 10 atomes de carbone et L est un groupe partant contenant un groupement anionique, l'acide conjugué du groupe partant ayant un pK_a dans l'intervalle de 6 à 13.

4. Composition selon l'une quelconque des revendications 1 à 3, dans laquelle la substance minérale hydratable est principalement constituée par un ou plusieurs sels d'acides ayant un pK_a dans l'intervalle de 1 à 9, de préférence de 2 à 7, mieux encore de 3 à 6,5.

5. Composition selon l'une quelconque des revendications 1 à 4, ayant un pH dans l'intervalle de 3,5 à 7, de préférence de 4 à 6,5.

6. Composition selon l'une quelconque des revendications 1 à 5, dans laquelle la substance minérale hydratable est choisie parmi le dihydrogènopyrophosphate disodique, le monohydrogénotripolyphosphate tétrasodique, le dihydrogènotripolyphosphate trisodique, le sulfate de sodium, le sulfate de magnésium, le bicarbonate de sodium, le tripolyphosphate pentasodique acidifié, le borax acidifié et les aluminosilicates acidifiés, et leurs mélanges.

7. Composition selon l'une quelconque des revendications 1 à 6, qui comprend en outre un acide organique choisi parmi les acides gras en C_8 — C_{24} , les acides polycarboxyliques, les amino acides polycarboxyliques, les acides polyphosphoniques, les amino-(acides polyphosphoniques) et leurs mélanges

8. Composition selon l'une quelconque des revendications 1 à 7, dans laquelle la substance minérale hydratable est principalement constituée par une ou plusieurs substances n'ayant pas de point de transition inférieur à 40°C.

9. Composition selon l'une quelconque des revendications 1 à 8, comprenant de 0,1% à 50%, de préférence de 1% à 45%, en poids, de précurseur d'agent de blanchiment, et de 25% à 99,9%, de préférence de 35% à 90%, en poids, de substance minérale hydratable.

10. Composition selon l'une quelconque des revendications 1 à 9, ayant une masse volumique apparente de 300 à 900 g/litre, de préférence de 400 à 750 g/litre et une granulométrie moyenne pondérale de 0,15 à 3 mm, de préférence de 0,5 mm à 0,14 mm.

11. Composition d'activateur de blanchiment selon la revendication 1, comprenant de 0,1% à 50% en poids du précurseur d'agent de blanchiment de type peracide organique soluble dans l'eau et de 10% à 99,9% en poids de la substance minérale hydratable soluble dans l'eau ou dispersable dans l'eau, la substance minérale hydratable étant principalement constituée par un ou plusieurs sels d'acides ayant un pK_a dans l'intervalle de 1 à 9.

12. Procédé de fabrication d'une composition d'activateur de blanchiment granulaire selon la revendication 1, comprenant la formation d'une suspension aqueuse du précurseur d'agent de blanchiment de type peracide organique soluble dans l'eau et de la substance minérale hydratable soluble

dans l'eau ou dispersable dans l'eau, l'ajustement du pH de la suspension (à 1% de solides) a une valeur dans l'intervalle de 3 à 8, de préférence de 3,5 à 7, et le séchage par atomisation de la suspension.

13. Procédé selon la revendication 12, dans lequel la suspension comprend de 30% à 60% en poids d'eau, de 0,05% à 35%, de préférence de 0,5% à 30%, mieux encore de 2% à 25%, en poids, de précurseur d'agent de blanchiment et de 10% à 69,95%, de préférence de 14% à 63%, en poids, de substance minérale hydratable.

14. Procédé selon la revendication 12 ou 13, dans lequel la suspension a une température de 60° à 90°C et est séchée par atomisation à une température d'entrée de l'air de 250°C à 350°C et à une température de sortie de l'air de 95°C à 115°C,

15. Composition de blanchiment comprenant de 5% à 99,5% en poids d'agent de blanchiment peroxygéné, caractérisée en ce que la composition de blanchiment comprend en outre de 0,5% à 95% en poids d'une composition d'activateur de blanchiment sous forme de granules séchés par atomisation selon l'une quelconque des revendications 1 à 11.

16. Composition de blanchissage comprenant de 25% à 99,5% en poids d'une composition de base de blanchissage comprenant de 2% à 40%, du poids de la composition de blanchissage, d'un tensioactif choisi parmi les tensioactifs anioniques, non ioniques, cationiques, ampholytes et zwittérioniques, et leurs mélanges, caractérisée en ce que la composition de blanchissage comprend en outre de 0,5% à 40% en poids d'une composition d'activateur de blanchiment sous forme de granules séchés par atomisation selon l'une quelconque des revendications 1 à 11.

17. Composition de blanchissage sous forme de granules séchés par atomisation comprenant, par rapport au poids de la composition, de 0,1% à 20% de précurseur d'agent de blanchiment de type peracide organique, de 2% à 40% de tensioactif choisi parmi les tensioactifs anioniques, non ioniques, cationiques, ampholytes et zwittérioniques, et leurs mélanges, et de 10% à 90% de substance minérale hydratable soluble dans l'eau ou dispersable dans l'eau principalement constituée par un ou plusieurs sels d'acides ayant un pK $_{\alpha}$ dans l'intervalle de 1 à 9, caractérisée en ce que le précurseur d'agent de blanchiment de type peracide organique est soluble dans l'eau et en ce que les granules ont un pH (en solution aqueuse à 1%) dans l'intervalle de 3 à 8.

18. Composition selon la revendication 16, comprenant de 15% à 90% en poids d'adjuvant de détergence minéral ou organique et de 5% à 35% en poids d'agent de blanchiment peroxygéné.

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